PB-237 809-T

USE OF ELECTRON MICROSCOPY FOR DETERMINATION OF THE GRANULOMETRIC DISTRIBUTION OF ATMOSPHERIC AEROSOLS WITH RADII OF BETWEEN 0.0035 MICROMETERS AND 1 MICROMETER

A. Renoux, et al

Environmental Protection Agency
Research Triangle Park, North Carolina

September 1974

DISTRIBUTED BY:

NTIS

National Technical Information Service
U. S. DEPARTMENT OF COMMERCE

312039

NTIS

EPA -TR 46-75

PB 237 809 -T

USE OF ELECTRON MICROSCOPY FOR DETERMINATION OF THE GRANULOMETRIC DISTRIBUTION OF ATMOSPHERIC AEROSOLS WITH RADII OF BETWEEN $3.5 \cdot 10^{-3}$ µm AND 1 µm [Determination de la repartition granulometrique, par microscopie electronique, des aerosols atmospheriques de rayons compris entre $3.5 \cdot 10^{-3}$ µm and 1 µm]

by

A. Renoux,[1] J.F. Butor[1] and G. Madelaine[2]
(1) Aerosols and Atmospheric Radioactivity Physics Laboratory, College of Sciences, Brest, and (2) Air Investigative Commission (CEA)-STEPPA, Fontenay-aux-Roses, France

Published in Chemosphere, 3(3), Pergamon Press, Great Britain, 1974, pp. 119-122.

Introduction

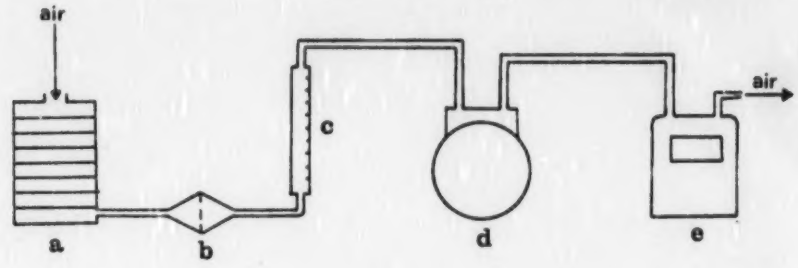
In a preceding article [1], we described a method for obtaining the granulometric distribution of aerosols with diameters in excess of 0.1 µm ($3 \cdot 10^{-5}$ cm). Now, there is another range which may be even more significant: that between 10^{-7} cm and 10^{-5} cm (in radius). So far these dimensions have been treated only theoretically. It therefore seemed essential to develop an experimental procedure permitting observation of aerosols with radii of a few 10^{-7} cm.

Translated for Technical Publications Branch, Information Services Division, EPA, from the French by Leo Kanner Associates, P.O. Box 5187, Redwood City, California 94063, (415) 365-3046, September 1974.

Reproduced by
NATIONAL TECHNICAL
INFORMATION SERVICE
U S Department of Commerce
Springfield VA 22151

Setup

Fig. 1 gives a schematic diagram of our assembly, which includes:



a. An eight-stage Andersen cascade impactor [2] with glass disks.

b. An impervious filter holder containing a nucleopore filter [3] with 0.4 µm openings, flattened against sintered bronze.

c. A rotameter calibrated for air at atmospheric pressure.

d. A Sartorius pump.

e. A Gallus type gas meter calibrated for air.

The rotameter c serves solely as a reference, since its readings are distorted due to the high loss of head arising from the Andersen impactor, the nucleopore filter and the sintered bronze. The true rate of flow, as well as the total quantity of air passing through the device, are given by the gas meter e. During our tests the rate of air flow was set at 28.3 ℓ/min^{-1}, corresponding to optimum operating conditions for the impactor.

Mode of Operation

Only the seventh and eighth stages of the impactor were studied by electron microscope, since the preceding stages trapped dust with diameters in excess of 2 µm. Copper grids for electron microscopy, collodionized and carbonized in a vacuum by a conventional process [4], were then placed on the glass disks of the seventh and eighth stages of the impactor. The deposit

on these disks was in the form of 216 spots (eighth stage) and 400 spots (seventh stage) distributed in concentric circles. The granulometry was found to be the same no matter which spot was selected, with the result that it was sufficient to use a very small glue point to glue six or seven copper grids immediately under the initial circumference of the metal screen of each of the stages examined.

Preliminary sampling was necessary to obtain an idea of the density of the deposit in order to avoid either an insufficient number of particles to permit valid statistical analysis, or, on the other hand, too high a concentration, producing agglomerates which would distort the measurements.

Samples were then taken in the following manner: a glass disk covered with grids was placed on the eighth stage, disks without grids being placed on the other seven stages of the impactor, and a nucleopore in the filter holder. Pumping was performed for between 2 and 4 min (the time being a function of the amount of dust collected). The eighth stage disk was then withdrawn and replaced by a disk without grids, while that of the seventh stage was replaced by a disk covered with grids, and suction was performed for 3 to 7 min. This seventh disk was then replaced by a disk without grids and pumping was performed for 30 min; the nucleopore should become slightly grayish, indicating an adequate dust concentration.

Sample Observation

The grids placed on the seventh and eighth stage disks were observed under the electron microscope after the diameter of the deposits was measured with an optical microscope. The electron microscope was used at its maximum acceleration voltage of 100 kV in order to obtain the best possible resolution, the magnification being set at 5000. (The magnification was calibrated by

means of a network replica with 1152 lines per mm.) A series of photographs of different areas of a complete deposit were taken, since the deposits were not uniform over their entire area.

To study the nucleopore filter it is necessary to make a carbon replica of this filter in a vacuum. The nucleopore is then dissolved by chloroform vapor, and the particles, or their imprint, remain on the carbon film. Carbon grids which have been placed under the nucleopore serve as a support for the carbon replica. Following this relatively lengthy operation (a few hours), these grids are "illuminated" by the electron beam, still at 100 kV, but with magnification of 15,000, and a series of 6.5 x 9 cm negatives are taken of various areas of the nucleopore. The particles are then counted and measured by means of a graduated eyepiece, the negatives being placed on a translucent plate lighted from beneath. Knowing the real surface area for a given photograph, the area of the entire deposit and the volume sampled, it is possible to derive the number of aerosols of given dimensions per cm^3. One thus obtains the granulometric distribution of atmospheric aerosols with radii of between $3.5 \cdot 10^{-3}$ µm and 2 µm.

Apparently only a small number of particles were destroyed by the electron bundle and thus escaped observation, since we used minimum illumination by a conventional procedure [5] and relatively low magnification. Specifically, it was easy to observe ammonium sulfate aerosols, which nevertheless are extremely sensitive to electron action, and were very numerous in our samples.

Results and Discussion

Fig. 2 shows a curve $dN/dR = f(R)$ obtained in our laboratory at Brest, a city with very little pollution, at a straight-line distance of 2 km from the ocean. The wind, being from the northeast, was a continental influence.

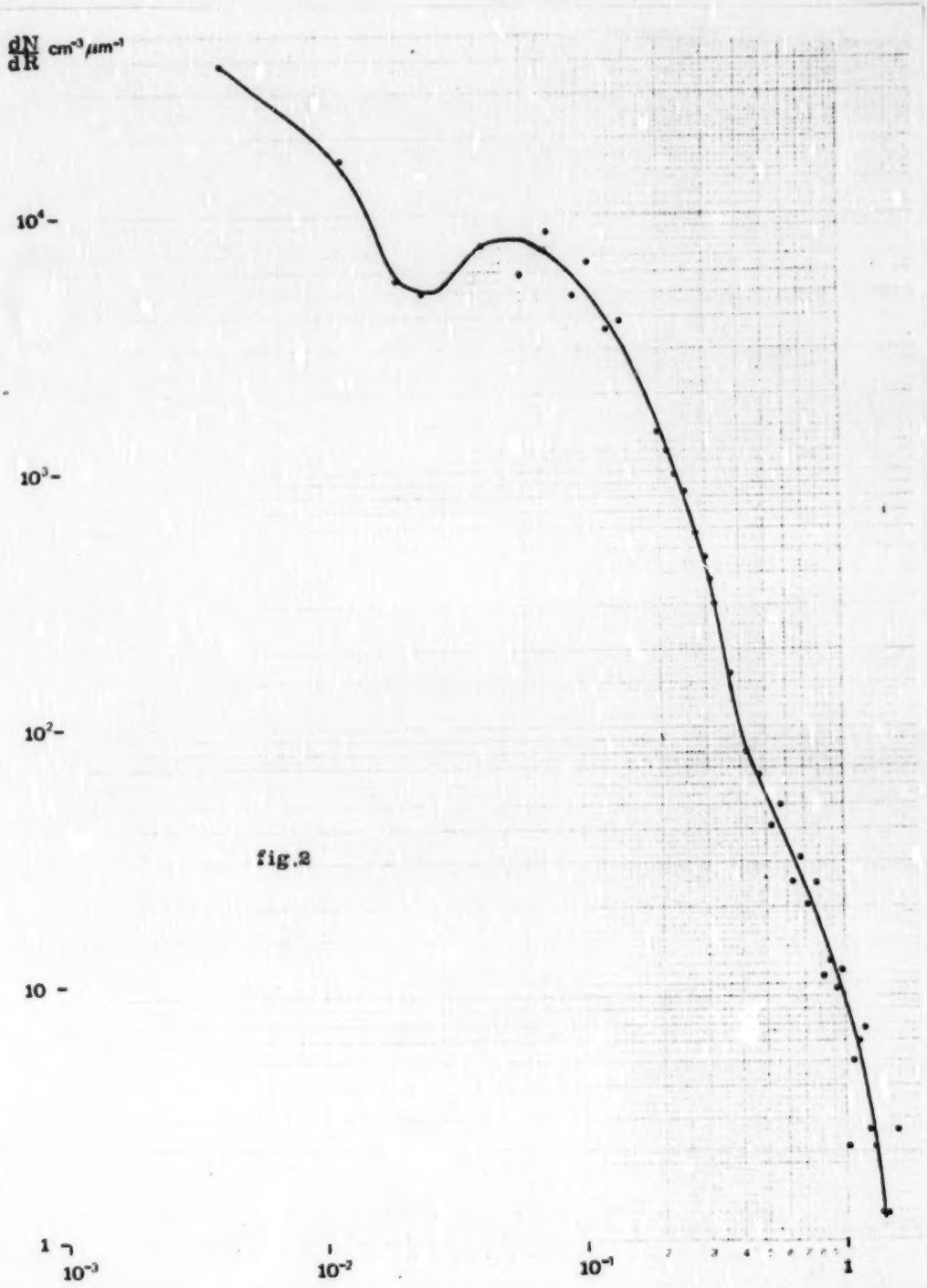


fig.2

Fig. 2.

Reproduced from best available copy.

A maximum may be observed for R = 5 1 6·10^{-2} m and a minimum toward R = 2.5·10^{-2} m, with an upward curve toward the finest particles. Our conclusion from this was that, despite the fact that the smallest aerosols very quickly become attached to the largest by heat coagulation, it does appear that, even in an unpolluted atmosphere, particles formed from gaseous impurities in the atmosphere (SO_2, NO_x, NH_3, hydrocarbons) play an appreciable role. This seemed to demonstrate the possibility, already analyzed theoretically by various investigators:

(a) of the continuous production of extremely small particles [6];

(b) of non-instantaneous fixation of these fine particles on the largest particles.

We are currently studying variations in this granulometric distribution as a function of meteorological conditions. In the case of a continental aerosol, the most frequent distribution is that shown in Fig. 2.

Bibliography

1. Renoux, A., and Tymen, G., forthcoming in Chemosphere (1974).
2. Air Sampling Instruments, American Conference of Governmental Industrial Hygienists, p. 023, 1972.
3. Spurny, K., Seminar on the Physics of Aerosols, under the direction of Prof. Bricard, Paris, 1969-70.
4. Renoux, A., and Butor, J.F., Pollution Atmosphérique, No. 58, 101 (1973).
5. Megaw, W.J., Seminar on the Physics of Aerosols under the direction of Prof. Bricard, Paris, 1969-70.
6. Report on the Study of Man's Impact on Climate. Massachusetts Institute of Technology, 1971.